



TITLE:

Studies on the Fluorometric Analysis. (XII)  
Fluorometric Determination of Aluminium  
by the Extraction of its Pontachrome Blue  
Black R Complex with Amyl Alcohol An  
Application to the Analysis of Pure  
Magnesium

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## ABSTRACTS

concluded that 0.01% is the optimum amount for nucleation from practical standpoint.

(2) **The influence of lithium content.** The glass of the composition, MgO 15, Al<sub>2</sub>O<sub>3</sub> 23, SiO<sub>2</sub> 62=100+XLi<sub>2</sub>O, where X=4, 6, 8, 12, by weight, was melted with and without addition of 0.01% platinum. The specimens of the same size as above were heated from room temperature to 1050-1100°C with the rate of 5°C/min., and then kept constant for one hour. The bending strength of platinum containing glasses increased remarkably when their lithium content was high (12%), whereas only a negligibly small increase was found in the glasses of low lithium content below 6%. This indicates clearly that the function of platinum was influenced by the amount of lithium in glass.

It was also confirmed that the glasses of low lithium content could be converted into a polycrystalline material of high mechanical strength even when no platinum was added. The investigation of this interesting phenomenon is now going on.

(3) **The optimum composition range in the system Li<sub>2</sub>O-MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>.** Keeping Li<sub>2</sub>O at a constant value of 12% other components were changed as; MgO  $x$ , Al<sub>2</sub>O<sub>3</sub>  $y$ , SiO<sub>2</sub>  $z$ , where  $x$ ,  $y$ ,  $z$  are mole ratio by weight and  $x+y+z=100$ , the range in which the two conditions, (a) glass may be obtained at a melting temperature lower than 1400°C, (b) the glass converts into the polycrystalline material without any noticeable deformation during the reheating will be satisfied. The range in which the condition (a) was satisfied was  $x$ : 0-30,  $y$ : 0-30,  $z$ : 60-100, and that of satisfying (b) was  $x$ : 0-20,  $y$ : 0-30 and  $z$ : 60-100.

## Studies on the Fluorometric Analysis. (XII)

### Fluorometric Determination of Aluminium by the Extraction of its Pontachrome Blue Black R Complex with Amyl alcohol An Application to the Analysis of Pure Magnesium

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*Nippon Kagaku Zasshi (Journal of the Chemical Society of Japan, Pure Chemistry Section), 81, 259 (1960)*

A fluorometric method for the micro-determination of aluminium based on the extraction of the Pontachrome Blue Black R complex with amyl alcohol was investigated.

Pontachrome Blue Black R reacts with aluminium to form a stable complex compound which shows an intense red fluorescence by ultraviolet light, with illumination and so the reaction has been used for the fluorometric analysis of aluminium. As the complex is easily extracted with amyl alcohol from an aqueous solution with pH 4.8-5.4, the sensitivity of the method can be increased and the influence of the quenching substances can be avoided, by the application of the solvent extraction.

The fluorescence of the complex in organic medium shows almost the same

## ABSTRACTS

energy spectrum as in aqueous solution (fluorescence band: 540–700  $m\mu$ , peak: 593  $m\mu$ ), and therefore a red filter which has the maximum transmission near 600  $m\mu$  is conveniently used as a secondary filter. Analytical procedure is as follows: To the sample solution, add 0.2 ml of 0.1% Pontachrome Blue Black R ethanol Solution and 5 ml of 10% ammonium acetate solution, dilute to about 50 ml with water and then adjust pH to 4.8. Heat in boiling water for 10 minutes, cool down to room temperature extract the complex formed with 25 ml of amyl alcohol, and measure the intensity of fluorescence of the organic layer.

By the procedure 0.05–1.5  $\mu g$  of aluminium can be determined. Copper, cobalt and titanium give negative error while gallium a large positive one. The effects of ferric iron and vanadate can be prevented by reducing with hydroxylamine.

The method is suitable for the micro-determination of aluminium because of its high sensitivity. Micro amount of aluminium (0.004–0.012%) in pure magnesium metal could be determined and good results were obtained.

### Spectrophotometric Method for Determination of Iron with Dibenzoylmethane

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Japan, Pure Chemistry Section)*, 81, 262 (1960)

A spectrophotometric method for the determination of ferric iron based on the extraction of ferric-dibenzoylmethane chelate with butyl acetate was developed.

The pure crystalline powder of ferric dibenzoylmethane chelate was obtained, and the formula was estimated as  $Fe[(C_6H_5CO)_2CH]_3$  from the iron content, 7.63% (theoretical 7.7%).

Ferric dibenzoylmethane chelate is soluble in organic solvents such as chloroform and butyl acetate. Absorption spectra of the chelate measured in chloroform and butyl acetate show two absorption bands in near ultraviolet region. The spectra of the chloroform solution, however, are unstable unless an excess of the reagent is present and are also affected considerably by the concentrations of the reagent. Therefore, the spectra of the chloroform solution are not suitable for the determination of iron. The chelate in butyl acetate medium shows absorption maxima at 320  $m\mu$  and 410  $m\mu$ , the former of which, however, can not be used for the determination because of the intense absorption band of dibenzoylmethane.

In the region near 410  $m\mu$ , the absorption spectra of the chelate extracted by the following procedure are the same as in the case above. Therefore the measurement of absorbances was made at 410  $m\mu$ . The effects of the reagent concentrations and pH values were investigated, and the analytical procedure was established as follows: To the sample solution with pH 2.5–3.5, 0.5 ml. of 5% dibenzoylmethane acetone solution is added and the solution is warmed in a water bath at 70°C for 10 minutes. The chelate formed is extracted with 20 ml. of butyl acetate, and the